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14. ABSTRACT

Verification and refinement of our understanding of mechanisms and their transitions at intermediate temperatures in the disk alloy Rene with the observation of microtwinning, continuous faulting and dislocation by-pass at successively higher temperatures above 650°C. Evid for the twin initiation process has also been obtained via TEM studies of specimens interrupted after small strain levels. A preliminary m for the novel microtwinning regime has been developed that appears to provide reasonable agreement with the present experimental c response for Rene 104 and Rene 88. Modeling at the *ab initio*, atomistic and phase field levels is providing important insight into the activ parameters associated with the observed deformation mechanisms, augmented by insight from 3D atom probe measurements on γ a composition and ordering. A novel phase field model of directional coarsening (rafting) during high temperature, low strss creep of blade a has ben developed. This model accounts for the local stress fields associated with matrix dislocations as well as the lattice misfit, demonstrates promising qualitative agreement with experiment. A model for creep of several "generation 4" single crystal blade alloys u "rafting" conditions has been developed.

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MEANS 2: MICROSTRUCTURE- AND MICROMECHANISM-SENSITIVE PROPERTY MODELS FOR ADVANCED TURBINE DISK AND BLADE SYSTEMS AFOSR GRANT # FA9550-05-1-0100

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Abstract

This effort has focused on verification and refinement of the mechanism transitions at intermediate temperatures in the disk alloy Rene 104, with the observation of microtwinning, continuous faulting and dislocation by-pass at successively higher temperatures above 650°C. Evidence for the twin initiation process has also been obtained via TEM studies of specimens interrupted after small strain levels. A preliminary model for the novel microtwinning regime has been developed that appears to provide reasonable agreement with the present experimental creep response for Rene 104 and Rene 88. Modeling at the *ab initio*, atomistic and phase field levels is providing important insight into the activation parameters associated with the observed deformation mechanisms, augmented by insight from 3D atom probe measurements on γ and γ' composition and ordering. Single crystals of Rene 104 have successfully been grown, and micro-tensile/compression as a function of crystal orientation will commence in the next year. A novel phase field model of directional coarsening (rafting) during high temperature, low stress creep of blade alloys has ben developed. This model accounts for the local stress fields associated with matrix dislocations as well as the lattice misfit, and demonstrates promising qualitative agreement with experiment. Finally, deformation mechanisms in high temperature creep of several "generation 4" single crystal blade alloys under “rafting” conditions have been studied and a model for creep under these conditions developed.

1. Research Objectives

This effort comprises a coordinated team of researchers from the Ohio State University (under Grant #FA9550-05-1-0135), University of Michigan (under Grant #FA9550-05-1-0100) and Johns Hopkins University (under Grant #FA9550-05-1-0102). The goal of the program is to develop improved models that will (a) incorporate more realistic representation of the relevant microstructures and micromechanisms, (b) enable modeling for a range of relevant service conditions (c) address time-dependent deformation in both disk and blade alloys, and (d) provide this information ev to the component design process, building upon the paths to the design process created in the DARPA Accelerated Insertion of Materials (AIM) program. This program is coordinated with Pro Tresa Pollock (University of Michigan) is leading efforts to characterize blade alloy microstructures and is providing single crystals materials for the rest of the program and Pro. Kevin Hemker (Johns Hopkins Unviersity) who is focusing on microtensile tests on single crystal variants of the disk alloy.

2. Accomplishments

• Completed assessment of mechanisms operative during creep in the two-phase superalloy R104 in the temperature range from 600-850°C. As a function of temperature, there is a transition from dislocation activity to microtwinning, extended/continuous stacking faults and dislocation climb by-pass at the highest temperatures.

• Completed detailed analysis of microtwinning mechanism, which is the dominant mechanism at the upper bound temperature for service of disk alloys in present applications. Microtwining, as well as the other precipitate shearing modes, is controlled by diffusion-mediated reordering.

• Determined the atomic-scale processes and activation barriers associated with reordering using the nudged elastic band method coupled with first principles calculations (VASP), which indicate that the diffusion coefficient for reordering should be similar to that for Ni-vacancy diffusion in the ordered $L1_2$ structure.

• Developed a phase field dislocation dynamics simulation which explains the transition from dislocation activity to microtwinning as a function of precipitate structure, applied stress and fault energies.

• Used Z-contrast imaging to detect or the first time segregation of heavy elements to microtwin and stacking faults in the precipitates after creep deformation. The fundamental role of these heavy elements (C, Mo and W) in modifying these fault energies has been explored using VASP calculations.

• Developed a model for high temperature creep under “rafting” conditions that is applicable to “Generation 4” type single crystal superalloys.

3. Program Summary and Present Status

During this period of the program, we have focused on improving our understanding of the mechanisms of creep in the polycrystalline disk superalloy, Rene 104, and more generally on developing the modeling tools necessary for treating critical issues in the high temperature response of both disk and blade alloys. These activities have benefited greatly from our interactions with industrial colleagues from GE Aircraft and Pratt-Whitney, and several collaborators from Wright Laboratories.

3.1 Mechanisms of Creep Deformation in the Disk Alloy Rene 104

Creep deformation studies have been conducted on the advanced disk superalloy Rene 104. Samples for creep experimentation were extracted from a fully heat-treated turbine disk supplied by GEAE and P&W. Previous results have shown that microtwinning is the principal deformation mechanism at elevated temperature (>650°C), which corresponds to the upper bound of service temperatures for this alloy [1,2,5]. As exemplified in Fig. 1, twinning proceeds by the passage of two, closely-coupled partial dislocations of the type 1/6<112>. When the partials encounter the γ' precipitates, high energy "complex stacking faults" (CSFs) are created in their wake due to the formation of unfavorable nearest-neighbor bonds. It is hypothesized that

movement of the twinning partials is controlled by elimination of the unfavorable bonds by local atomic rearrangements (reordering as illustrated in Fig. 1) and is responsible for the temperature and rate dependence exhibited by these alloys. Other distinct deformation mechanisms also reported at higher temperature involving precipitate shearing may also be controlled by reordering as they all involve shearing of the γ' precipitates. The kinetics associated with

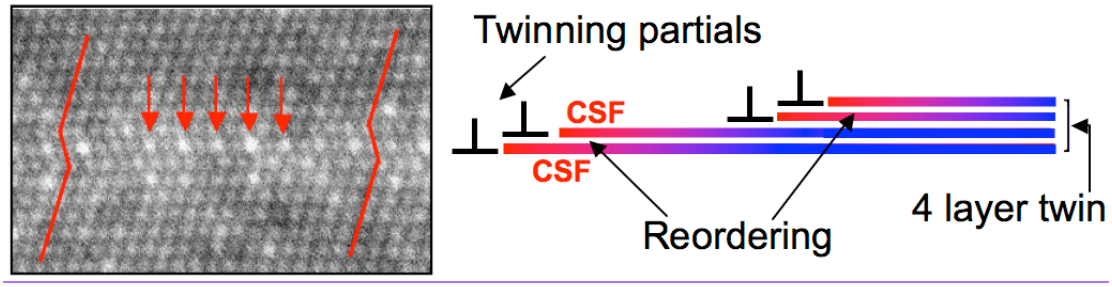

Fig 1: (a) HAADF-/STEM image of two-layer nanotwin with evidence of heavy element segregation (bright columns indicted by arrows) and (b) hypothetical reordering-controlled twinning mechanism in the γ' phase.

reordering have been explored using VASP, and initial results indicate that Ni-vacancy diffusion and reordering should have similar diffusion coefficients [13].

Several heavy elements such as Mo, Ta and/or W are added to commercial alloys since they tend to improve high temperature strength, but the reason for this improvement has not been understood in detail. Evidence of segregation to fault and microtwin boundaries has also been obtained using HAADF-STEM imaging, as indicted in Fig. 1. Interestingly, segregation is only observed in the γ' particles [13]. Thus, the elements may be swept to the interfaces in the course of the reordering, ultimately impeding microtwin thickening, and thereby providing enhanced creep strength.

3.2 Parametric Studies of Mechanism Transitions Using Phase Field Dislocation Dynamics

Insight into the microstructural factors affecting the transition from matrix dislocation activity to microtwinning have been obtained in recent experiments coupled with phase field dislocation dynamics modeling [11]. We have observed that under creep (677ºC and 724MPa and microstructure (fast cooling/fine microstructure) conditions for which microtwining is the dominant process at 2% strain, a distinctly different substructure is observed at smaller strain levels. Detailed dislocation indicates that ½<110> matrix dislocations, all having the same Burgers vector in a given grain, dissociate widely into Shockley partial dislocations that have a Burgers vector of the type a/6<112>. The leading partial dislocations appear to have "percolated" between the secondary γ' precipitates, leaving them unsheared. While the process whereby this structure subsequently evolves into microtwins is presently not clear, the decorrelation of ½[110] matrix dislocations nevertheless appears to be a necessary precursor to microtwinning.

The interaction of a/2<110> dislocation with precipitates and the various kinetic pathways associated with their dissociation have been explored using the so-called microscopic phase-field (MPF) model. The MPF enables treatment of complex γ′ shearing processes without any a priori

assumptions about dislocation geometry, core structure, and formation of stacking faults. The result of one such simulation for a single screw-oriented dislocation as it interacts with a secondary γ′ precipitate population with a size (diameter) of 300 nm and an interparticle spacing of 75nm is shown in Figure 2.

As the applied stress in this case is below the Orowan stress for the perfect dislocation (AB), it is sufficient to drive the leading partial (Aδ) into the channel because of the smaller line tension (which is approximately proportional to $b^{2,}$, where b is the Burgers vector of the partial). Note the stress resolved on the trailing partial is only half that on the leading partial for the orientation chosen. As shown in Fig. 2a, the leading Shockley sweeps out and creates an intrinsic stacking fault in the matrix while the trailing partial remains at the entrance to the γ channel. For the same microstructure, but increasing the matrix stacking fault energy 10 fold to 100mJ/m^2, leads to a quite different result, as shown in Fig. 2b. Under these circumstances, when the dislocation makes its way to the confines of the γ channel, due to the increase in matrix stacking fault energy, the dislocation does not dissociate into Shockley partials. Instead, the dislocation remains intact as a full a/2<110> and is immobilized.

These preliminary results of the phase field model demonstrate several important factors that trigger the decorrelation of the ½[110] dislocations, and leading ultimately to deformation via microtwining. These factors include the matrix stacking fault energy, as high fault energy inhibits decorelation, and ultimately microtwinning. At a stress below the Orowan bowing stress for ½[110] dislocations, decorrelation will be favored for microstructures in which the spacing between secondary particles is smaller. If the stress is in excess of the Orowan bowing stress, then the matrix dislocations remains undissociated, regardless of stacking fault energy. Note also that the absence of twins at this higher stress level would be difficult to understand if dislocation activity and microtwinning were operative in parallel. The phase field simulations instead suggest that if the microstructure and stress conditions are favorable to ½[110] matrix activity, rather than decorrelation, then microtwining will not be operative. The orientation of the applied shear stress relative to the operative Burgers vector should have a significant role on decorrelation. This indicates that decorrelation and microtwinning may only be favored over an relatively narrow range of applied shear stress orientation. These attributes will be incorporated into unique constitutive formulations that are being developed for the crystal plasticity modeling by Ghosh.

3.3 Deformation Mechanisms and Microstructure Evolution for Rafted Microstructures

Our modeling efforts have been coordinated with deformation studies at University of Michigan by Prof. Tresa Pollock, also supported by the MEANS2 program. As is typical of the single crystal blade alloys, directional coarsening of the γ' precipitates occurs during creep exposure. Dislocation substructures are being studied at various stages of creep deformation to obtain critical information on rate-limiting mechanisms in the rafting regime. A ground-breaking phase field model that reproduces these remarkable morphological changes has also been developed under this program in collaboration with Prof. Yunzhi Wang and his graduate student, Ning Zhou, and initial results of this model are described below and in [6,8]. The simulation technique is based on an integrated phase field model that for the first time incorporates simultaneously spatiotemporal evolution of both precipitate morphology and dislocation

structures. The initial configurations consisting of cuboidal γ′ particles and dislocations in γ channels are constructed according to experimental observations and phase field simulation of dislocation filling process in the γ channels. The spatial variation of chemical potential of solute atoms is evaluated based on local concentration and stress and diffusion fluxes in different γ channels are analyzed.

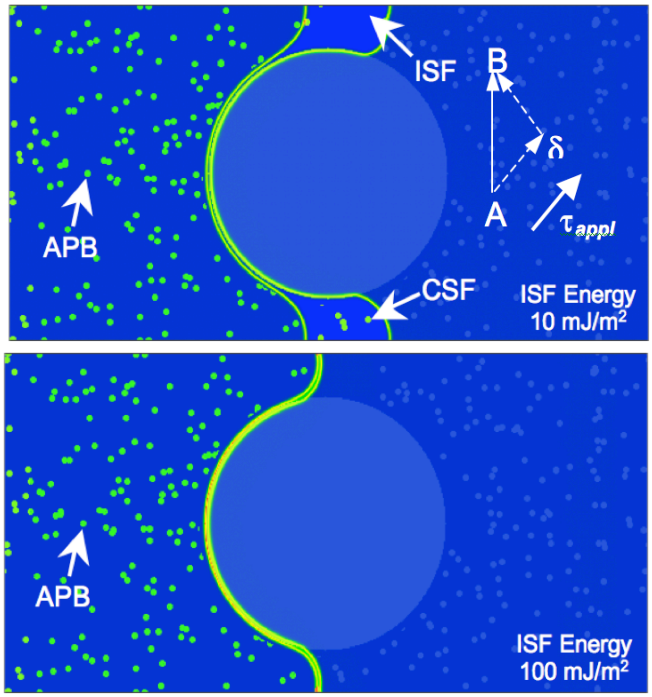

Figure 2. Phase field simulation of a/2<110> dislocation (AB) interaction with a secondary γ′ precipitate at an applied shear stress of 115MPa, 300nm γ′ precipitate size, and 75nm γ channel width spacing. The simulation shows the effect of tertiary γ′ precipitates and stacking fault energy a) 10mJ/m^2 ISF and b) 100mJ/m^2 ISF energy w/tertiary γ′. Periodic boundary condition is applied in the vertical direction. Note the spontaneous dissociation under stress of the matrix dislocation as it squeezes through a narrow matrix channel.

In this model, dislocation motion in γ channels is driven by the local stress. This local stress includes contributions from lattice misfit, the external load, and misfit dislocations at the γ/γ′ interface. Simultaneously, the diffusion-mediated evolution of microstructure is determined by both chemical driving forces and elastic interactions with dislocations, as well as the effect of lattice misfit. Through this integrated approach, rafting of the γ' phase has been predicted under various conditions of misfit sign and loading type, and the results agree with our experiments and other analyses. Some results are shown in Figs. 3 and 4. It has also been found that modulus mismatch is not a necessary condition for rafting, as has been assumed previously in the elasticity-based models for rafting. Under the homogeneous modulus assumption (ignoring the modulus mismatch between γ and γ′ phase), the simulation results show rafting of γ′ precipitates is closely related to the type and distribution of dislocations at the γ/γ′ interfaces. The sign of lattice misfit and the direction of the applied load determine the dislocation configurations in the γ-channels and the rafting directions. It is found that dislocations prefer normal channels (normal to the direction of applied load) for a positive misfit alloy under compression, and a negative misfit alloy under tension which later develop N type rafting, while parallel channels were preferred by dislocations in a positive misfit alloy under tension and a negative misfit alloy under compression which develop P type rafting.

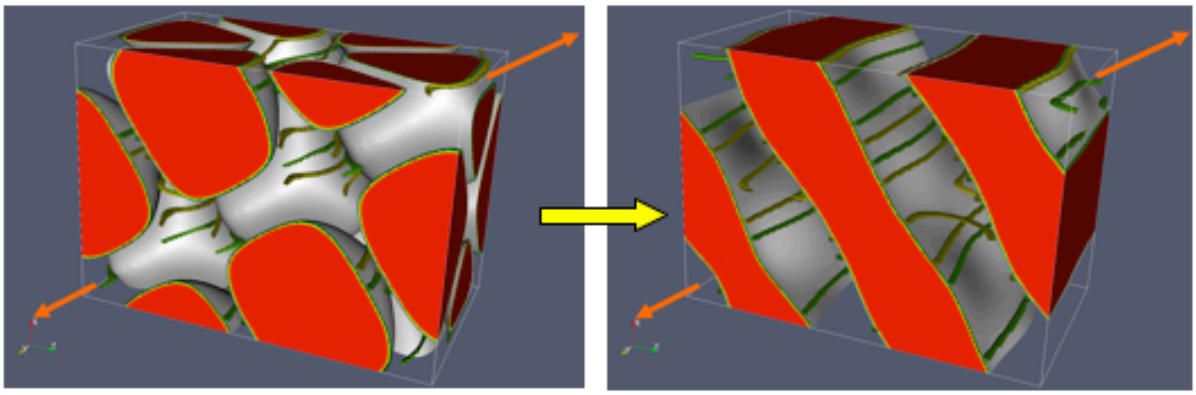

Figure 3: Phase field model result showing N type rafting for alloys with -0.3% lattice misfit under 152 MPa tensile stress along [001] after 11 hours.

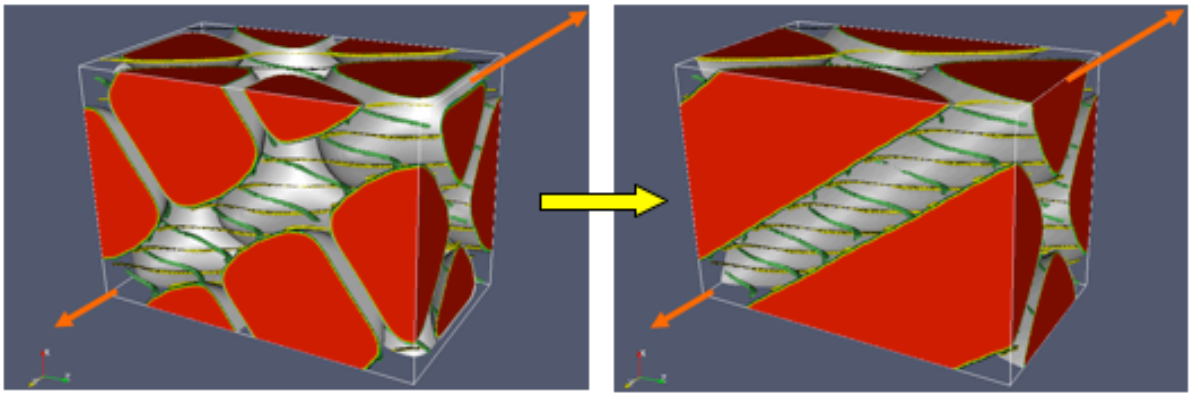

Figure 4: Simulation for P type rafting for alloys with +0.3% lattice misfit under 152 MPa tensile stress along [001] after 7 hours.

The model has further been developed to include modulus mismatch, using an effective transformation strain to describe the channel plasticity. A parametric phase field simulation study [12,14] has resulted in the following conclusions:

• When both channel plasticity and modulus mismatch are considered, the type of rafting is determined solely by the direction of the applied stress and the sign of the lattice misfit (i.e., the sign of $\frac{\sigma^{ex}}{\delta}$). The sign of the modulus mismatch does not change the rafting type, which is true even when the difference in the shear modulus is about 15%, i.e., $\frac{(C_{11}-C_{12})^{\gamma}}{(C_{11}-C_{12})^{\gamma'}} = 85\%$.

• The P type rafting completes faster than the N type rafting. The reason is that for the P type rafting, solute atoms flow from two vertical channels to one horizontal channel leading to a rod; while for the N type rafting, solute atoms flow from one horizontal channel to two vertical channels leading to a plate. In other words, forming a plate takes longer time than forming a rod.

• Modulus mismatch between precipitate and matrix does alter the rafting kinetics. If the precipitate phase has higher elastic stiffness than that of the matrix, the rafting process will take

less time to complete as compared to a system having softer precipitates (assuming the same applied stress, misfit and dislocation density).

• Dislocation density will also affect the kinetics of rafting. The higher the channel dislocation density, the faster the rafting process. The density of dislocations is determined by the magnitude of the applied stress, lattice misfit and the starting microstructure (γ′ particle size and γ channel width).

3.4 Creep Deformation of Advanced Single Crystals in the Rafting Regime

The alloys studied cover a range of composition representative of advanced "Generation 4" single crystals. In these alloys directional coarsening of the γ' precipitates occurs during creep experiments conducted at 950˚C and 295 MPa. Dislocation substructures are being studied at various stages of creep deformation in sections parallel to the applied tensile stress to obtain critical information on rate-limiting mechanisms in the rafting regime. No comprehensive creep models have been developed for this stress / temperature regime, which has received relatively limited attention beyond studies that address the directional coarsening phenomenon. With regard to the rate-limiting mechanisms three possibilities are being considered: (1) creep controlled by viscous glide and climb of dislocations across the γ' rafts; (2) creep controlled by viscous glide and climb in the matrix γ phase and (3) creep controlled by diffusion-limited reactions at γ - γ' interface that produce dislocations for shearing of the γ' rafts.

Alloy	Ni	Al	Ru	Ta	Re	W	Co	Cr
UM F20	67	13.9	3.5	2.8	1.5	1.0	2.5	8.0
UM F30	59	13.8	3.5	2.7	1.5	1.0	10.5	8.0

Table 1: Compositions for two experimental "generation 4" alloys under investigation.

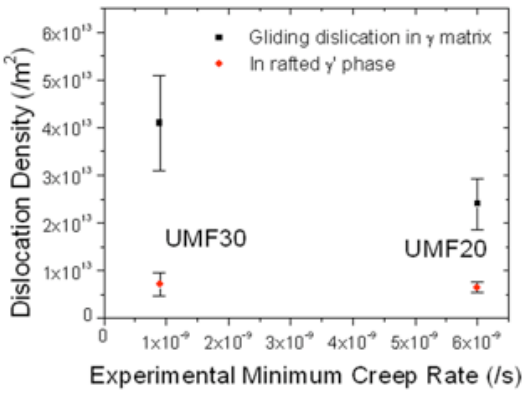

Figure 5: Measured dislocation densities in the γ and γ' phases after creep in rafted microstructures.

The most recent studies have focused on two alloys designated UM-F20 and UM F-30, whose compositions are similar except for the Co level and are listed in Table 1. We note the presence of dislocations in the γ' rafts, at the γ - γ' interfaces and in the gamma matrix. The density of dislocations that appear to be mobile in each of the phases was measured, and the results are shown in Figure 5. Note that with changes in minimum creep rate, there are changes in the matrix dislocation density, but no changes in the density within the precipitates. Furthermore, the creep rate decreases in UMF30 compared to UMF20, with increasing dislocation density in the matrix and also with increasing Co in the matrix. This seems to indicate that dislocations accumulate in the matrix until favorable conditions develop at the interface for pairing of dislocations in a configuration that will result in shearing of the γ' rafts. Based on the experimental observations reported here, two possibilities for the rate controlling mechanism during high temperature creep of superalloys in the rafting regime can be envisaged: (1) the

viscous motion of dislocation pairs through the γ' rafts and (2) periodic (time dependent) penetration of dislocations from the interfacial networks into the rafted precipitates. Srinivasan and coworkers have suggested that pairwise cutting can occur within ' rafts by a combined climb and glide mechanism in CMSX-6 at 1020°C and 80 MPa [44]. The creep rate is then limited by the viscous motion of the climbing dislocation through the raft. In this case, the experimental creep rate coincided well with the calculated strain rate, based on a model of the viscous shearing. A similar calculation was performed for the present Ru-containing experimental superalloys using the climb velocity determined by Srinivasan and coworkers:

$$\dot{\gamma} = 0.5\rho b v_c$$

where is the dislocation density, b is the Burgers vector, and v_c is the climb velocity determined for CMSX-6 as $4x10^{-8}$ m/s. For the present Ru-containing alloys, the climb velocity is assumed to be similar based on a lower creep temperature but higher magnitude of the externally applied stress. The density of dislocations within the rafts, , was determined from TEM images taken longitudinally in the gage section of UM-F20 after creep rupture, and was determined to be $3x10^{12}$ /m2. Therefore, the creep rate is estimated as 1.510^{-5} /s. The experimentally determined creep rate for UM-F20, however, was several orders of magnitude lower, at $6x10^{-9}$ /s.

The considerable difference between the calculated and experimentally-determined creep rate suggests that the viscous motion of dislocation pairs through the γ' rafts is not the rate limiting process for this alloy at 950°C and 290 MPa. The other possibility for the rate limiting creep mechanism is the time dependent penetration of the dislocations through the interfacial dislocation networks and into the rafts. The time dependence may be a result of either (a) the frequency at which two parallel a/2<110> dislocations arrive at the γ' interface, or (b) the frequency at which a gliding matrix a/2<110> dislocation encounters a compatible a/2<110> network dislocation. The former is unlikely based on the present TEM observations that show excess dislocations present in the matrix channels. Rather, the present microscopy studies suggest that the time dependence of penetrating pairs of dislocations is the most likely controlling mechanism for creep in this class of alloys at the conditions evaluated. The network structure is rapidly established along the γ' interfaces during primary creep; further entry of dislocations that sustain the creep process would elicit interactions between the existing network structure and the gliding matrix dislocations. Considering the deposition of a dislocation segment by glide of a matrix a/2<110>, it is likely that its position at the interface will lie between two pre-existing network dislocations. If the dislocation is of the correct (similar) sign, climb of this deposited dislocation onto an adjacent glide plane with a network segment would provide the necessary pairwise-cutting source for raft penetration. Note that significant climb has already occurred within the network to achieve a desirable misfit-reducing configuration of the net. Another local climb event of significance would involve dislocations of opposite signs climbing to an adjacent plane in order to annihilate the network dislocation, resulting in a reduction in dislocation density and localized coarsening of the network. Because dislocations entering a channel with an established network have an equal chance of being of similar or opposite sign to neighboring dislocations, the rate of production of a dislocation pairs that can shear rafts would be directly related to the rate of coarsening of an established network during creep.

Considering that the creep rate is limited by the climb velocity of dislocations into critical shearing configurations at the scale of the interfacial network, then the overall creep rate can be estimated from the following:

$$\dot{\varepsilon} = b\rho\frac{d\bar{x}}{dt}$$

where is the interfacial dislocation density and dx/dt represents the average glide distance per raft shearing event per unit time. The dx/dt term will be governed by the climb distance, λ and climb rate, vc, of the dislocations at the γ' interface, as well as the width of the raft, β. Thus:

$$\frac{d\bar{x}}{dt} = \frac{\beta}{\lambda}v_c$$

with climb velocity as

$$v_c = \frac{D\Omega\sigma b}{b^2 kT}$$

where D is the diffusivity, is the atomic volume, b^3, k is Boltzmann's constant, and T is the temperature [45]. Thus the creep rate would then be

$$\dot{\varepsilon} = \frac{\rho D\sigma b^3}{kT}\left(\frac{\beta}{\lambda}\right).$$

It is necessary to consider only the density of dislocations involved in generating shearing pairs of dislocations from the interfacial network. Thus only a fraction of the dislocations in the νετωορκ will experience an interaction with the appropriate type of gliding dislocation; this fraction is designated by a pre-exponential factor, Z,

$$\dot{\varepsilon} = \frac{Z\beta\rho D\sigma b^3}{\lambda kT}.$$

Recent interdiffusion experiments on Ni-Re binary alloys suggest that the interdiffusion coefficient at 950°C is approximately D = $6\text{x}10^{-18}$ m^2/s [46]. The density of interfacial dislocations is approximately 10^{15}/m2, while β/λ = 10 for the present alloys. The stress is approximately 290 MPa in the present experiments. Considering a creep rate of approximately 10^{-8} /s, an estimate of the pre-exponential factor, Z, on the order of 10^{-6} is obtained. Not

surprisingly, this suggests that only a few segments of the dislocations within the interfacial network are involved in the accumulation of strain during high temperature creep. So it is worth considering the spacing between such active links in the network present with density act. With $Z = 10^{-6}$, approximately one dislocation out of every 10^6 of the network dislocations is active. This spacing between active links should be of the same order of magnitude as observed in Figure 6 and the spacing that is calculated from the density of dislocation pairs in the γ' rafts, 10^{12} /m^2, corresponds to approximately 1 dislocation pair shearing the raft per 1 μm. The fraction of active dislocations was determined from the experimental spacing between shearing events and the interfacial dislocation spacing as approximately $5*10^{-4}$, quite similar to the calculated value, Z, which was approximately 10^{-6}. Thus, this model is physically reasonable. One implication is that a high resistance of the γ' phase to shearing is important to improving creep properties. An alloy that has a high precipitate shearing resistance will accumulate excess matrix dislocations. A high precipitate shearing resistance will, in turn, be dependent on precipitate composition and the associated APB and/or complex fault energies. Finally, creep resistance can be improved with the addition of elements with slow interdiffusion coefficients in the matrix, in order to slow the climb kinetics in the near interfacial regime. Rhenium additions are well known to be effective in this regard.

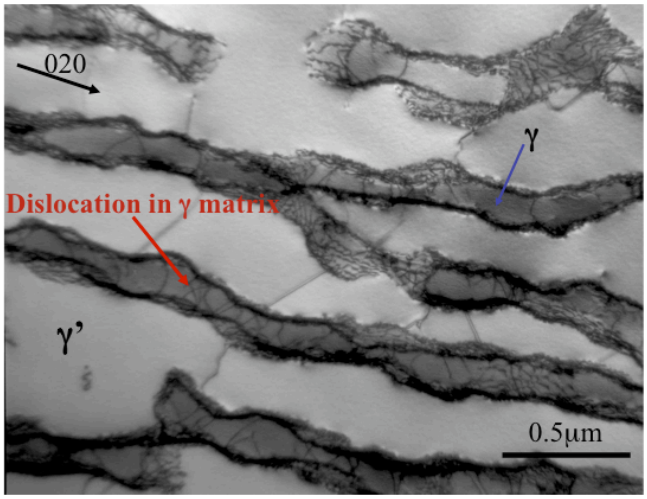

Fig. 6 – Dislocations occasionally shearing rafts in 5.7 Ru - 6.7 Cr - 2.4 Co, after creep at 950C/295MPa.

Personnel Supported at UM During Reporting Period

Laura Carroll	Graduate Student	Materials Science and Engineering
Shuwei Ma	Postdoctoral Researcher	Materials Science and Engineering
Tresa Pollock	Professor	Materials Science and Engineering

UM Publications

S. Ma, L. Carroll and T.M. Pollock, "Development of γ Phase Stacking Faults during High Temperature Creep of Ruthenium-Containing Alloys, *Acta Materialia* 55, 5802, (2007).

L.J. Carroll, Q. Feng and T.M. Pollock, "Interfacial Dislocation Networks and Creep in Directionally Solidified Ru-Containing Nickel-Base Single Crystal Superalloys", *Metallurgical and Materials Transactions* 39A, 1290, (2008).

N. Zhou, C. Shen, P.M. Sarosi, M.J. Mills, Y. Wang and T. Pollock, "γ′ Rafting in Single Crystal Blade Alloys – A Simulation Study," submitted to *Materials Science and Technology*, 2008

Transitions

Extensive discussions and exchange of information has already occurred with our industrial partners at GE Aircraft Engines (Dave Mourer, Mike Henry and Deb Whitis) and Pratt-Whitney (Michael Savage). Characterization and modeling of creep mechanisms are serving as a basis for understanding low cycle fatigue in similar disk alloys in the NASA Propulsion 21 program. Laura Carroll is now employed at GE Aviation and is involved alloy development for turbine airfoils.